Making the Most of APS Enhancements in Sector 2

Friday, 5 April 2002

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Experimental Facilities Division

Synchrotron Radiation Instrumentation
Collaborative Access Team

Micro-Techniques Group (SRI-CAT/Sector 2)

MISSION

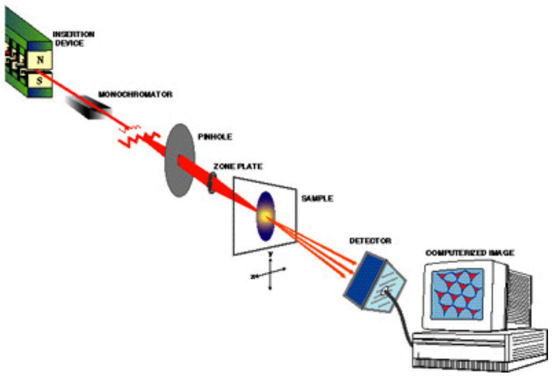
Development and application of instrumentation and techniques for high resolution x-ray microscopy.

Personnel

9 Physicists
2 Postdocs
2 Engineers
3 Technicians
1 Grad Student

SCOPE

Material, biological, and environmental science at dedicated beamlines in the 0.5-4 and 5-35 keV energy regions.

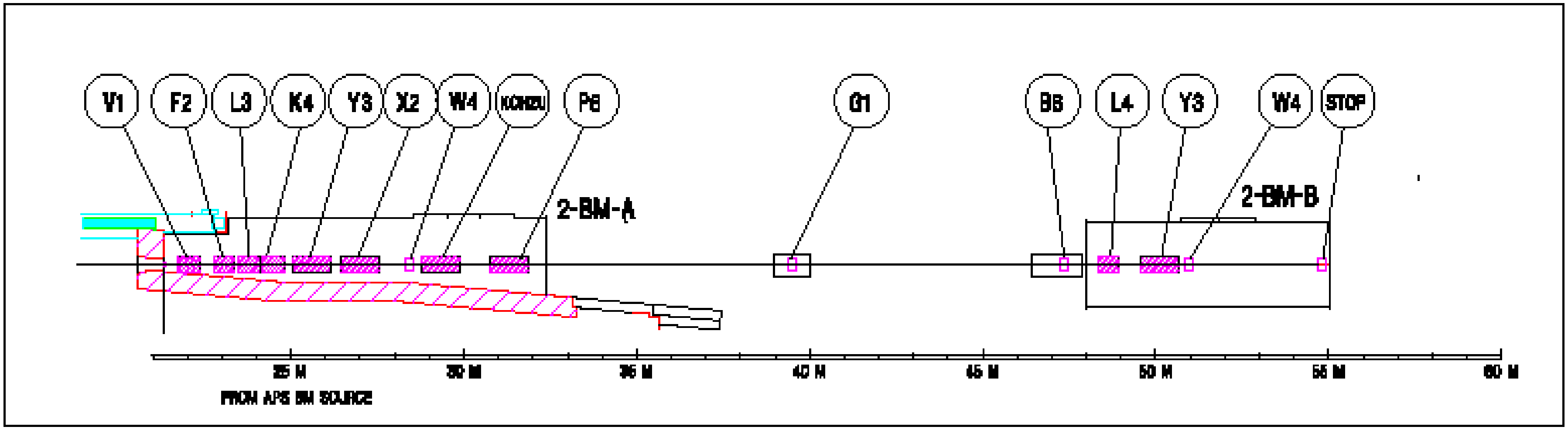


Sector 2 programs

• **X-ray microscopy**
  • Transmission
  • Fluorescence
  • Microdiffraction
	2-BM, 2-ID-B, 2-ID-D

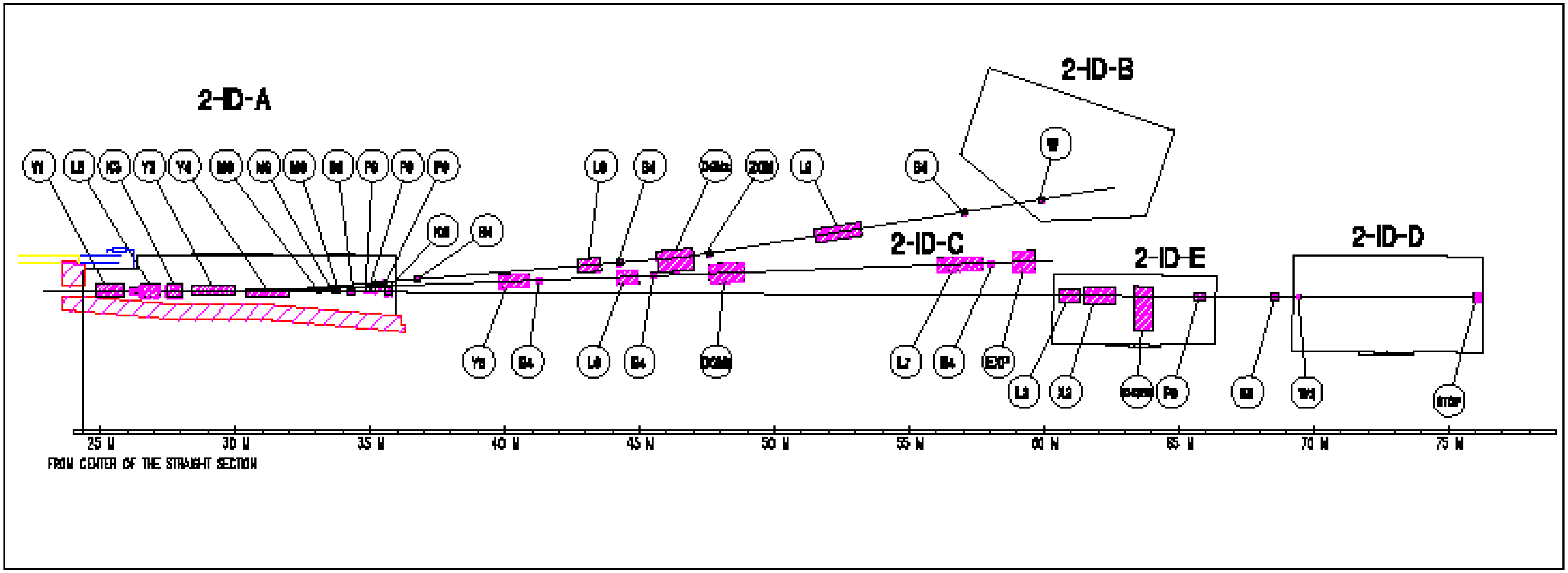
• **Coherent x-ray scattering**	**2-ID-B, 2-ID-D**
• **Deep x-ray lithography**	**2-BM-B**

SRI-CAT Beamline 2-BM



APS

SRI-CAT Beamline 2-ID



X-ray microscopy

- **Provides wealth of information**
  - Transmission — measure electron density
  - Fluorescence — measure elemental distribution
  - Spectroscopy — extract chemical information
  - Tomography — reveal 3D structure
  - Diffraction — reveal structure, strain
- **Image the internal structure of thick or optically opaque objects**
- **No special sample preparation (staining, drying, sectioning)**
- **Non-destructive, quantitative, *in vivo, in situ***

Recent applications

Biological/biomedical

- Uptake of Pt anti-cancer agents in ovarian cancer cells (D. Phillips)
- Microbiology of bacteria (K. Kemner)
- Role of Fe and K in mycobacteria infection (D. Wagner)
- DNA photocleavage by TiO_2 in mammalian cells (G. Woloschak)
- 3D micro-anatomy of crustacean crania (M. Fahnenstock)

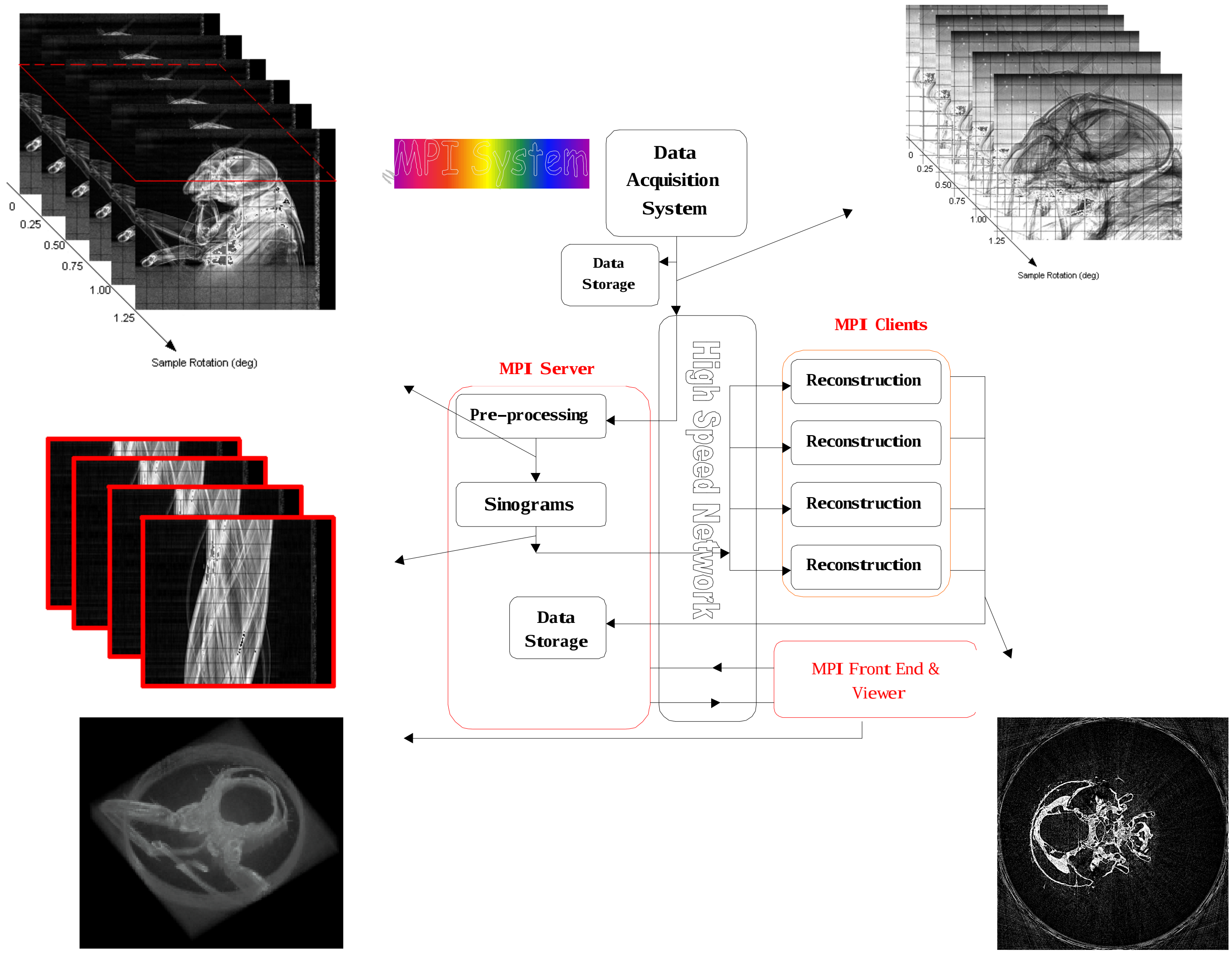
Environmental

- Elemental makeup of particles in air-borne pollutants (D. Cohen)
- Study of plant-fungi symbiotic relationship (S. Pratt)

Materials science

- Microstructure of membrane protein crystals at room temperature (M. Caffrey)
- 3D study of electromigration voids in interconnects (Z. Levine)
- 3D fluorescence study of nuclear fuel shell (G. Ice)
- Study of antiferromagnetic domains in Cr (P. Evans)

Fast x-ray microtomography at 2-BM



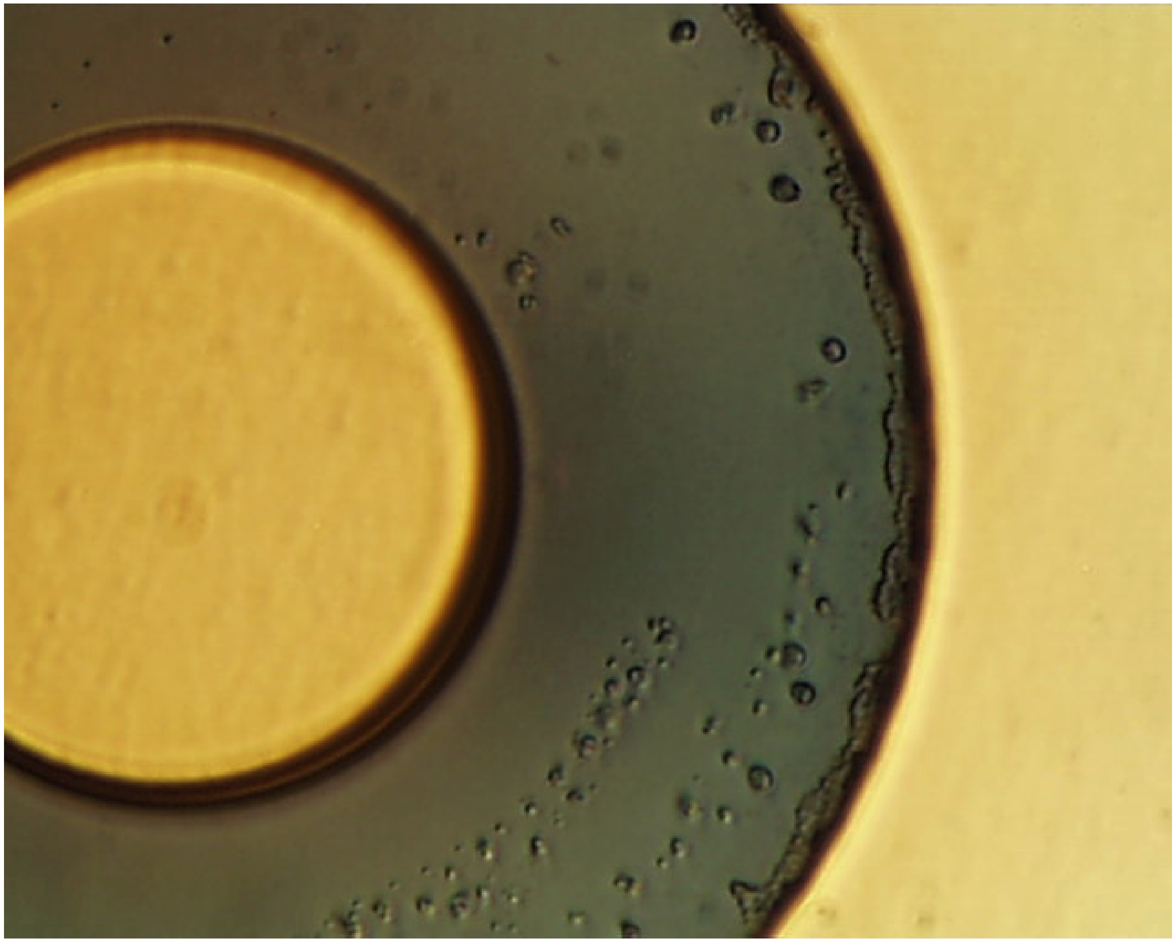
Y. Wang, et al., *Rev. Sci. Instrum.* 72, 2062 (2001)

Micro/nano focusing capabilities

- Zone plates provide best resolution compared to K-B optics and refractive lenses

- World-class performance of Sector 2 x-ray microscopes
- Development of key supporting instrumentation, methods
- Supporting other APS beamlines at existing and new CATs
  - Instrumentation design (e.g., Nano-CAT)
  - zone plates (in use by 6 CATs)
- No other beamlines (yet) optimized for these capabilities in SRI-CAT or at APS

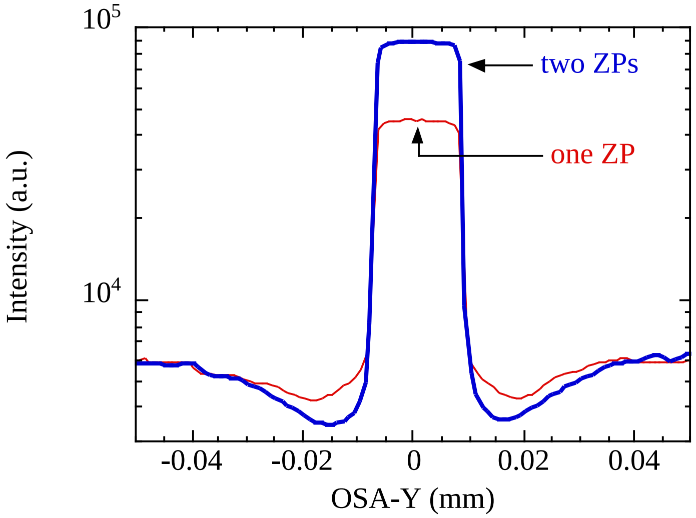
Highest resolution zone plates to date



δ_r = 40 nm, R = 49 μm, t = 0.13 μm, Ni
f = 5.7 mm and η = 3.0% at 1.8 keV

APS

Improving zone plate efficiencies



Relative performance of one zone plate vs. two stacked zone plates with outer zone width of 100 nm, at 8 keV.

Efficiency	**12% @ 8 keV (0.8 μm Au)** **33% @ 8 keV (1.6 μm Ni)** **39-45% @ 8 keV (3-step blazed)** **25% @ 40 keV (2 stacked)**
Focus-to-background	**3.6×10^3 (3-step blazed)**

True x-ray lens!

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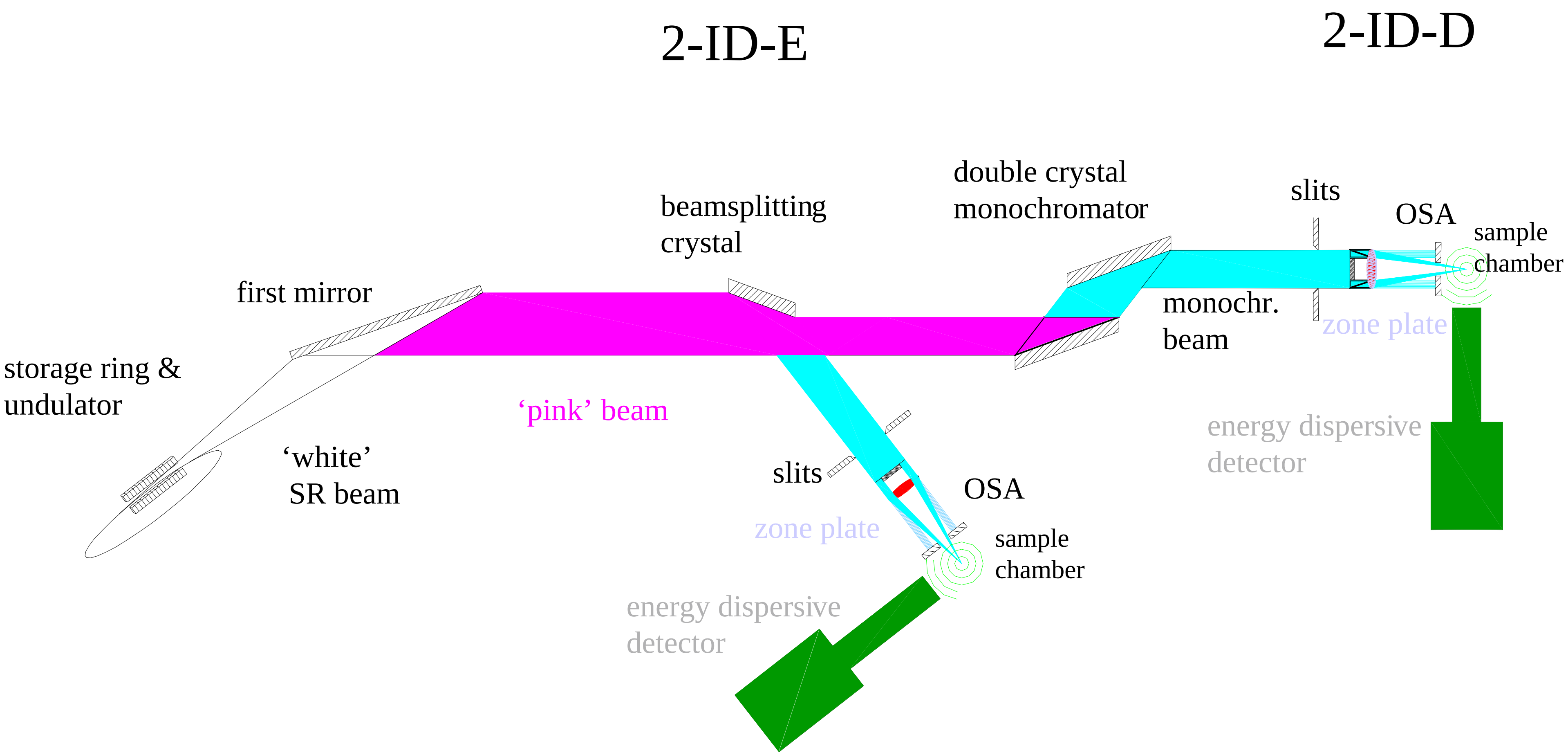
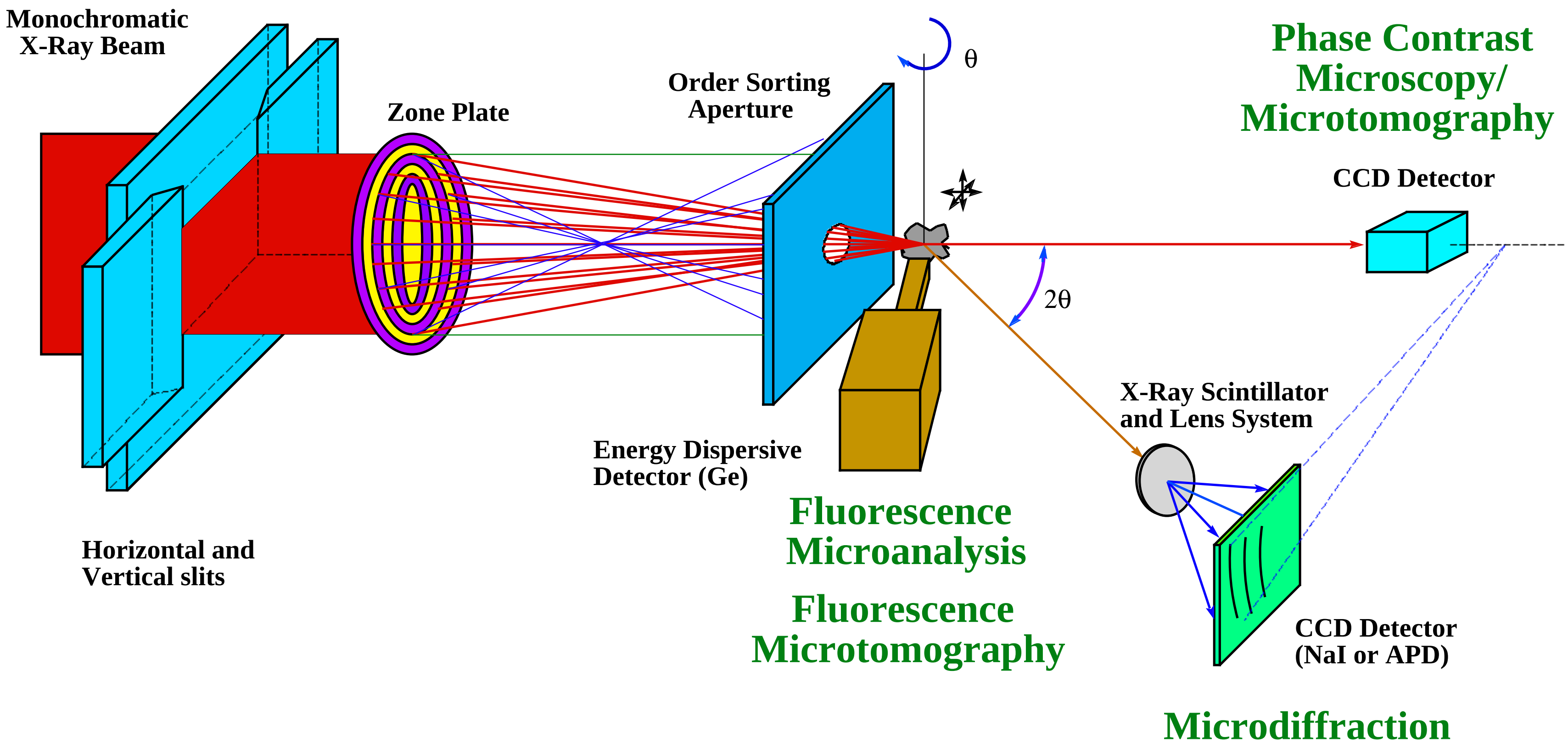
2-ID-D/E hard x-ray microscopy beamline

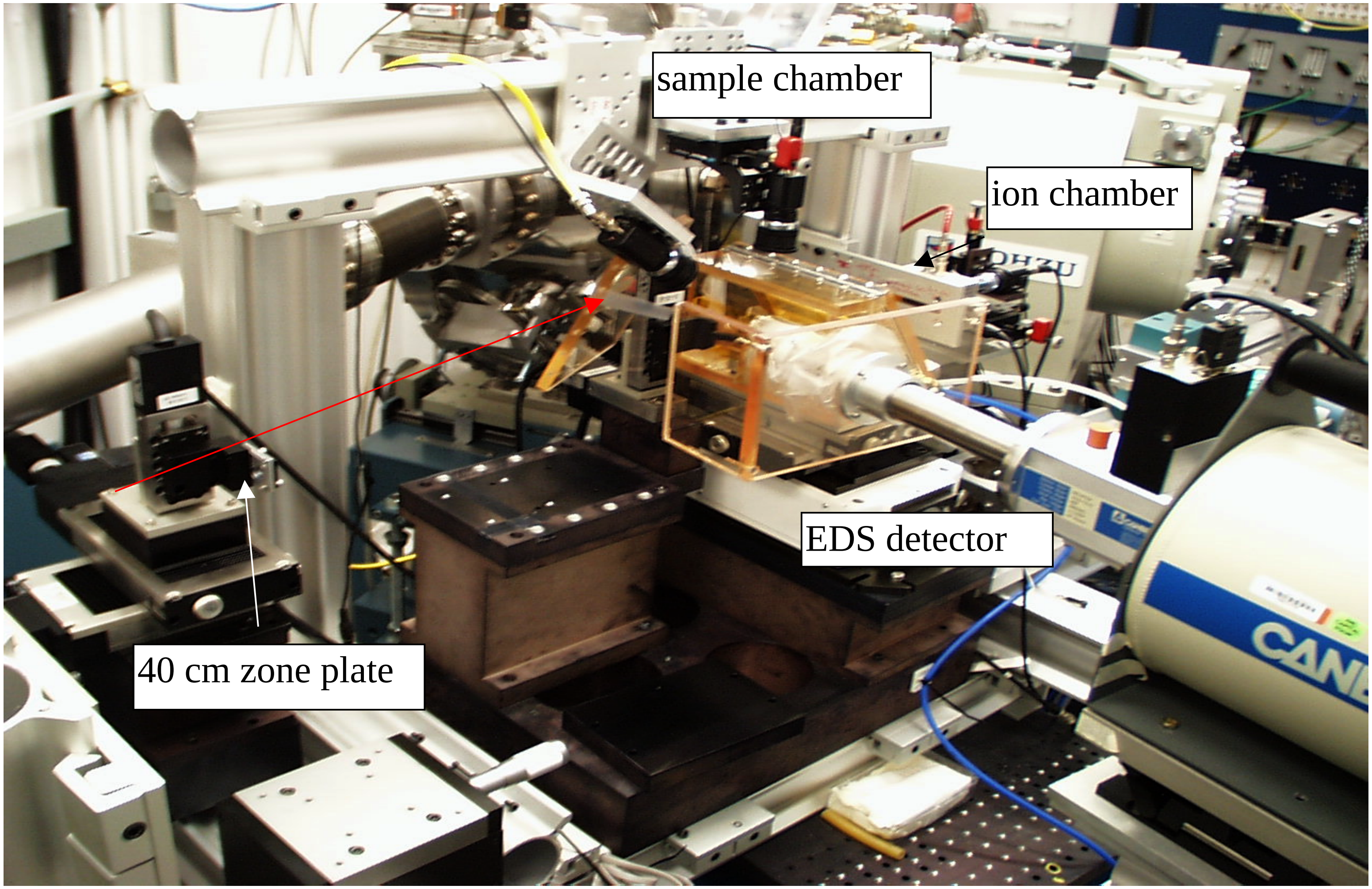


(only fluorescence setup shown for 2-ID-D)

2-ID-D hard x-ray (5-12 keV) microprobe



2-ID-E side-branch XFM



Downstream-outboard view (beam enters from left)

X-ray fluorescence microscopy

Intrinsic advantages compared to charged particles

- 10-10^3 times higher fluorescence cross sections
- 10-10^5 times better signal-to-background ratios
- 10^{-3}-10^5 times less radiation damage for same elemental detectability

  Crucial for biological samples
- Better spatial resolution for samples with thickness >1 μm
- Selectively excite one element versus another
- Better sensitivity for chemical state and local environment
- Simpler sample preparation

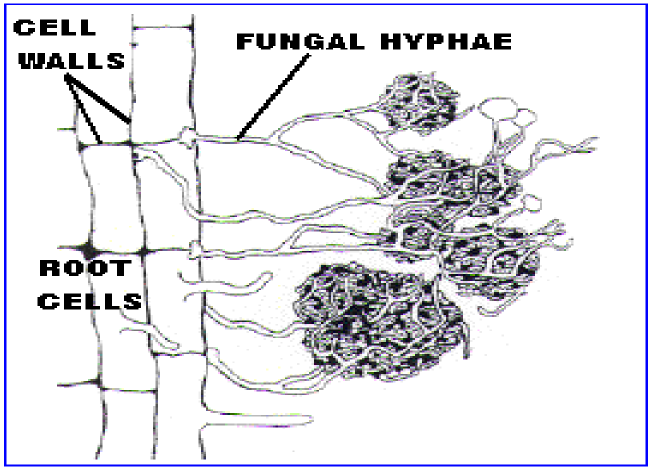
  Study biological samples in theirnatural hydrated state

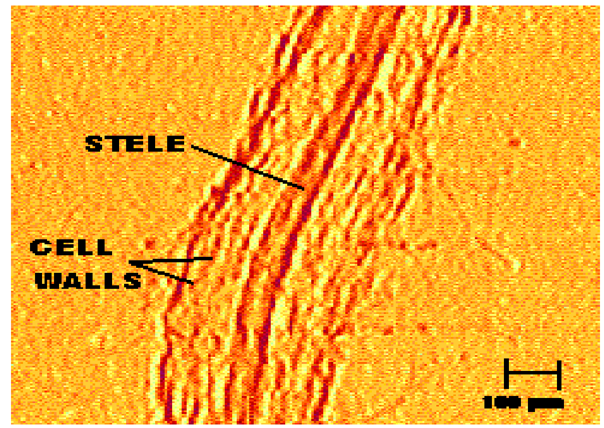
Demonstrated capabilities

- 10-100 ppb elemental sensitivity (100 ppm and 10 ppm for EIXE and PIXE)
- 30 attogram of trace element detection sensitivity
- Micro-XANES of Mn at 3ppm concentration

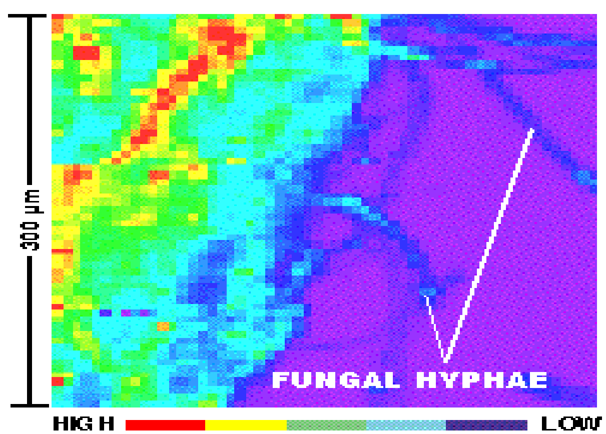
X-RAY IMAGING STUDIES
OF THE MYCORRHIZAL FUNGUS-PLANT SYMBIOSIS

90% of the world's plants, including essentially all economic crops, make use of mycorrhizal associations with fungi

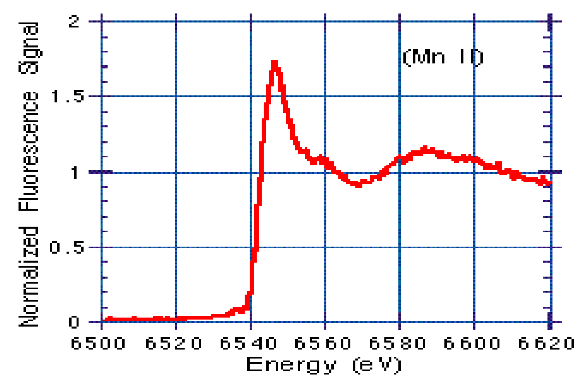




Mn-FLUORESCENCE IMAGE
OF PLANTAGO ROOT



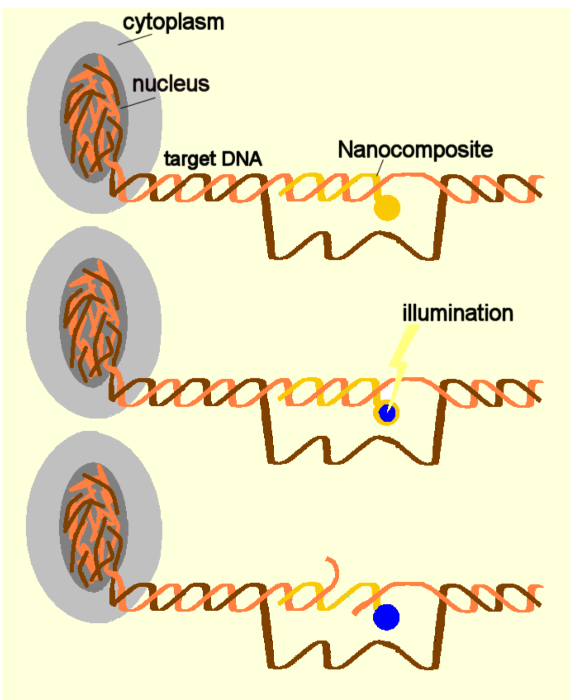
MICRO-XANES OF Mn

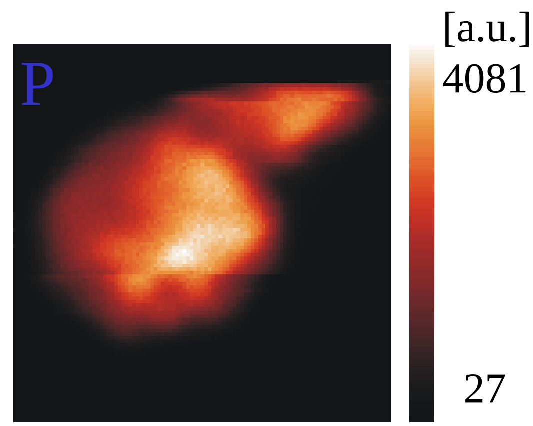


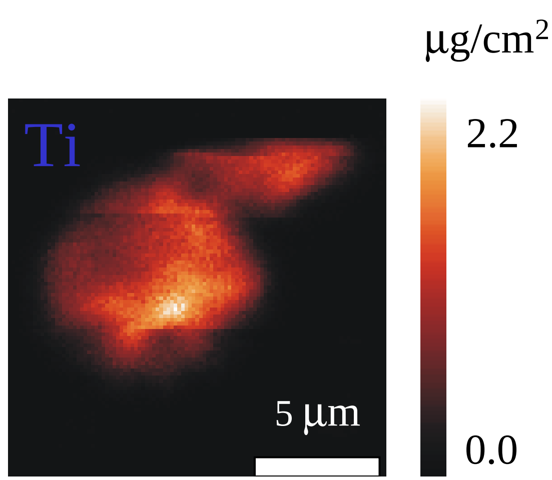
Z. Cai, K. Kemner, B. Lai, H. Lee, M. Miller, S. Pratt, W. Rodrigues, W. Yun

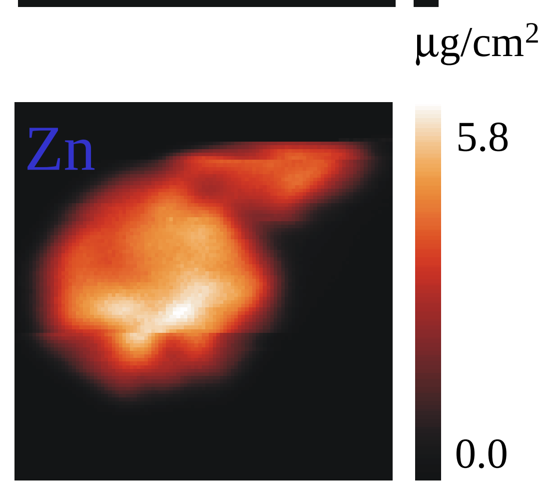
TiO_2-DNA nanocomposites in mammalian cells

- Cell is transfected with TiO_2-DNA nanocomposites
- DNA targets specific chromosomal region
- TiO_2 photocleaves DNA strands upon illumination
- Potential use in gene therapy
- Map Ti distribution using X-ray induced K_α fluorescence, to quantify success rate of TiO_2-DNA transfection and visualize target
- Affinity of transfected DNA to ribosomal DNA causes nanocomposites to localize to the nucleolus

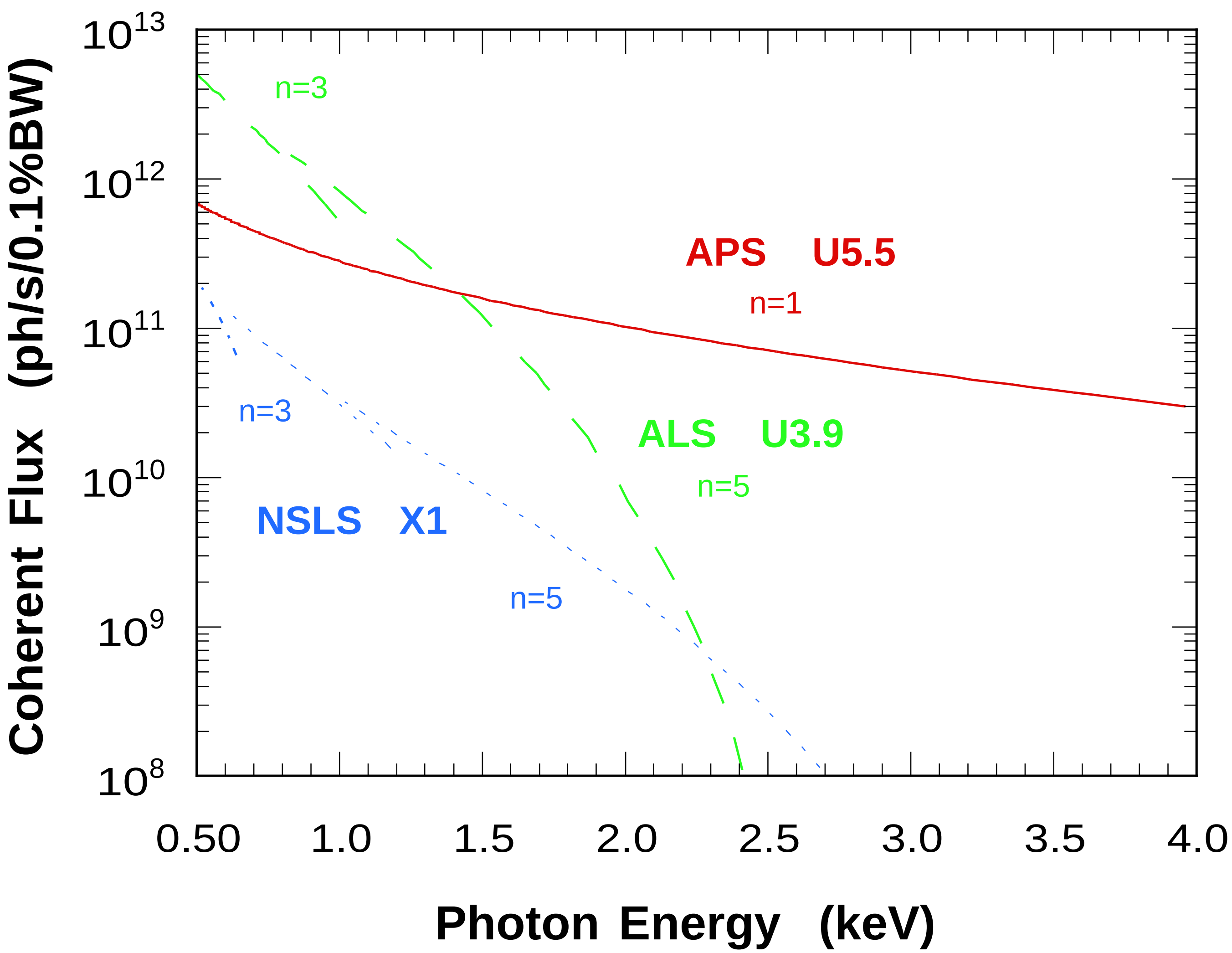




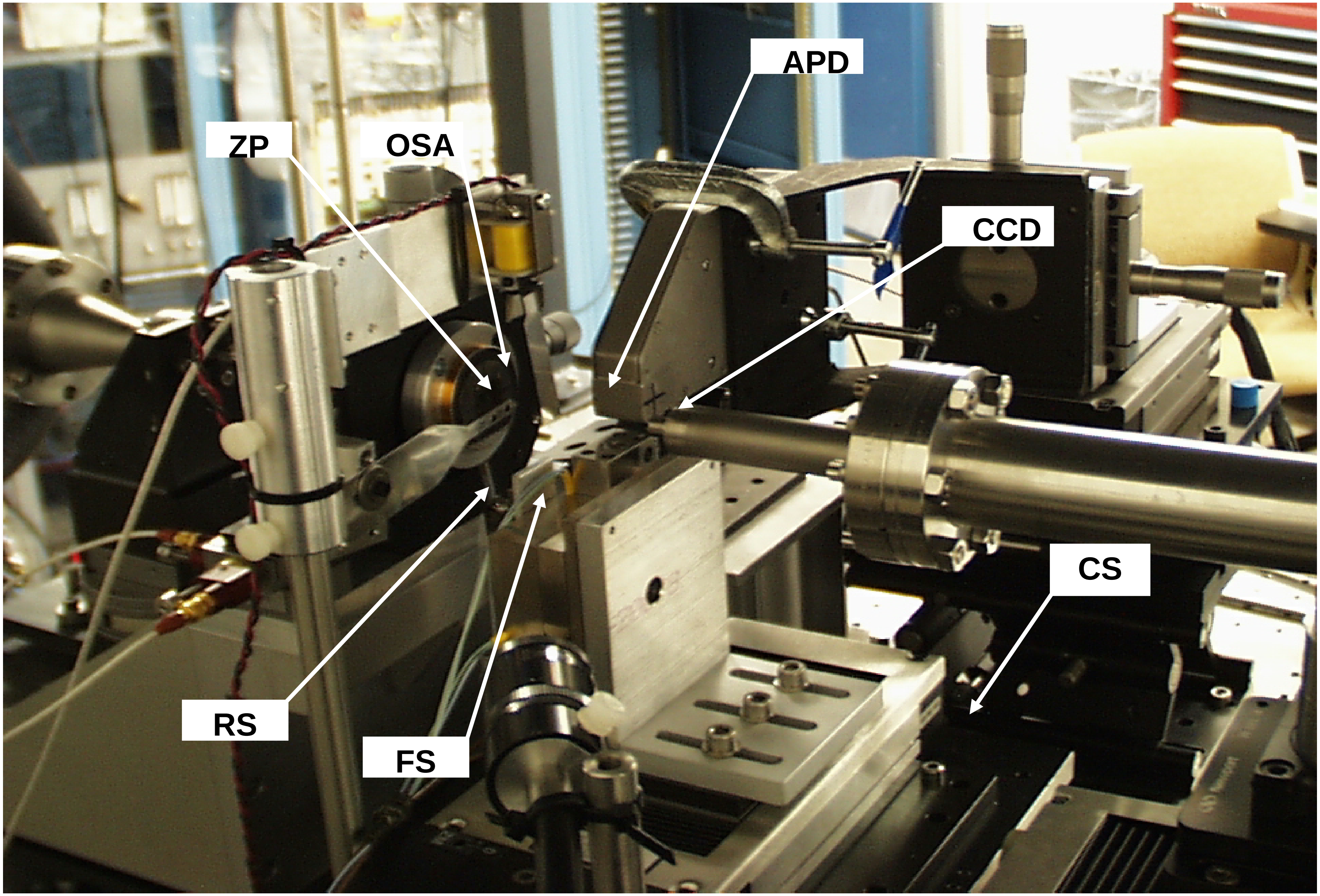




Spatially coherent flux in the 1-4 keV region



2-ID-B soft x-ray (1-4 keV) scanning microscope

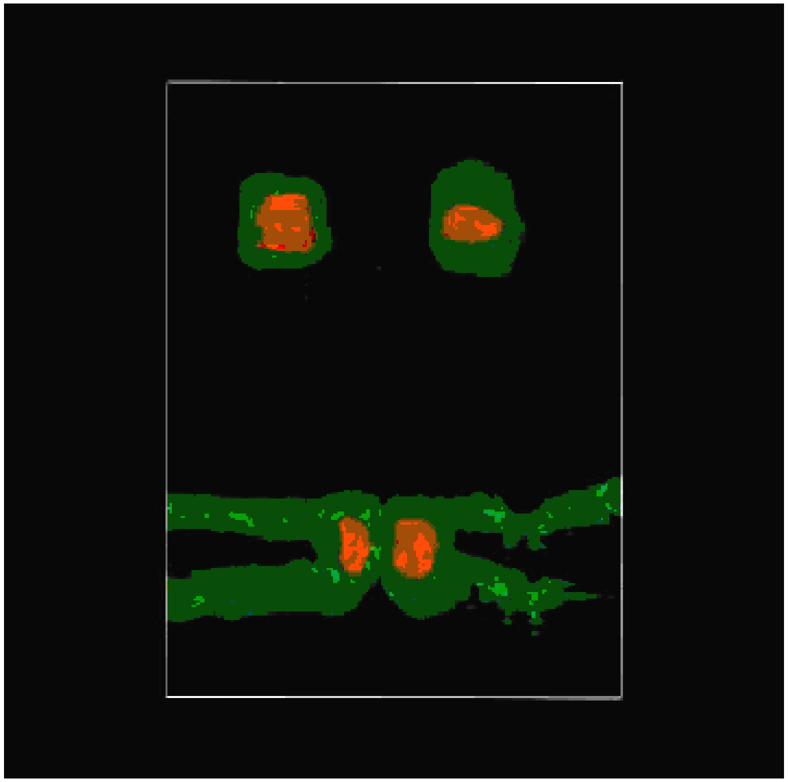


Fast scan-on-the fly capability

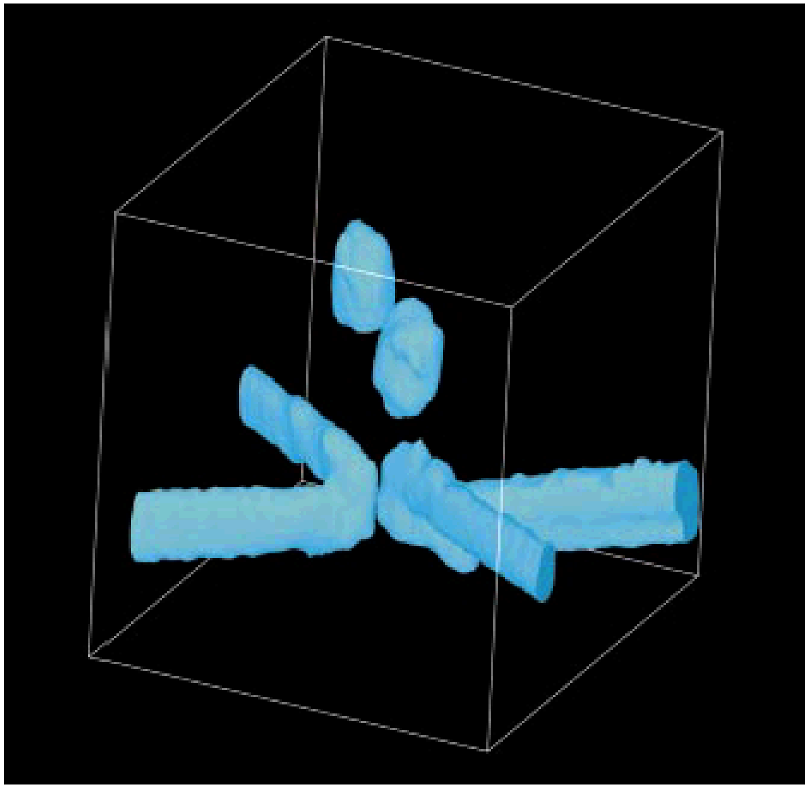
- Position-based "on-the-fly" scanning now routine
- Use multiscannel scaler, advance channel with position steps
- Clock-normalization recovers exact dwell time
- Scan speed — 2 ms/pixel — stepper stage
  200 µs/pixel — piezo stage
- Target — 10 µs/pixel

10^9 ph/s $\Rightarrow$ 1% statistics/pixel

Scanning nanotomography of Al/W/Si chips



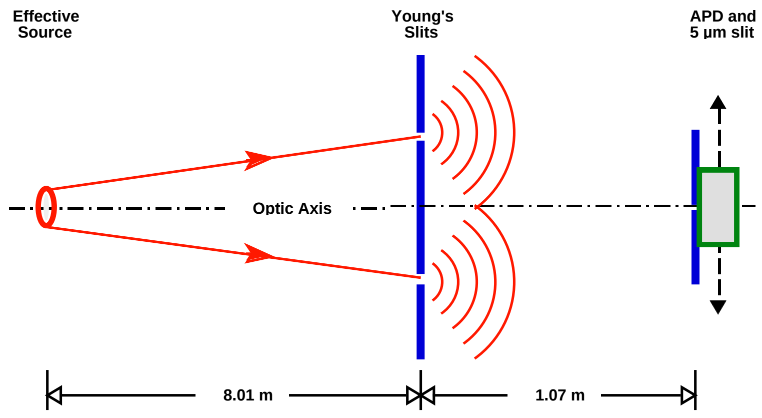
SIRT reconstruction of 13 STXM projections (±69°, 57 nm steps, 1573 eV) through two-level sample. Al interconnects (green) are joined by W vias (red). Two FIB markers are at top of image. This in-plane (side) view cannot be observed directly.



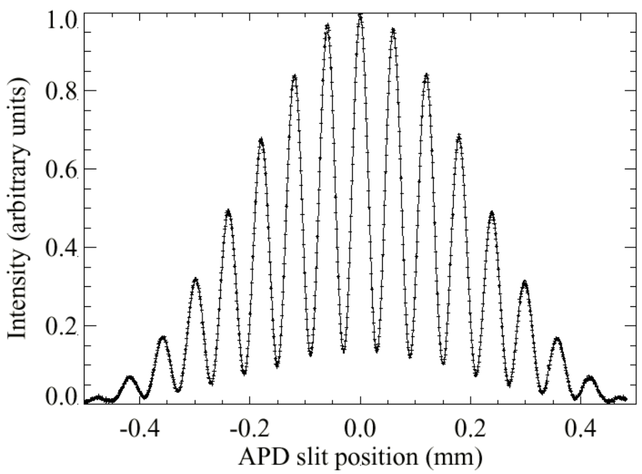
Bayesian reconstruction using same data. FIB plane to upper interconnect plane distance is 5.4 ± 0.5 μm.

Z. Levine, et al., *Appl. Phys. Lett*. 74, 150 (1999)

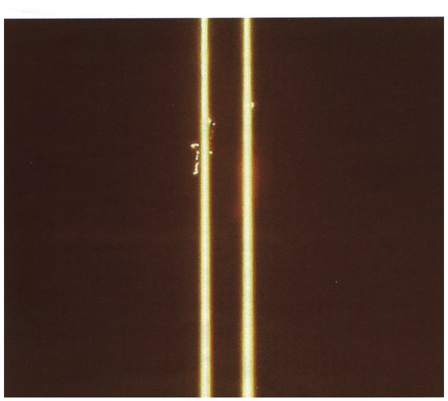
Coherence measurement of undulator radiation



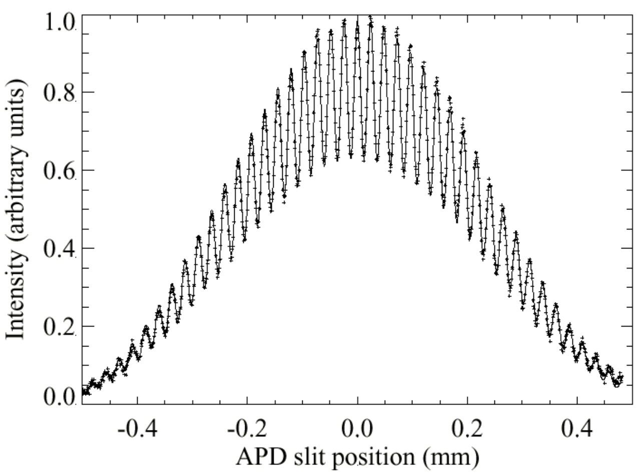
Young's experiment (top view) at 2-ID-B beamline With E = 1.1 keV, 50 µm entrance slit, ~200 µm exit slit, and Young's slit separations of 10-200 µm.



Young's fringes with 20 µm slit separation.



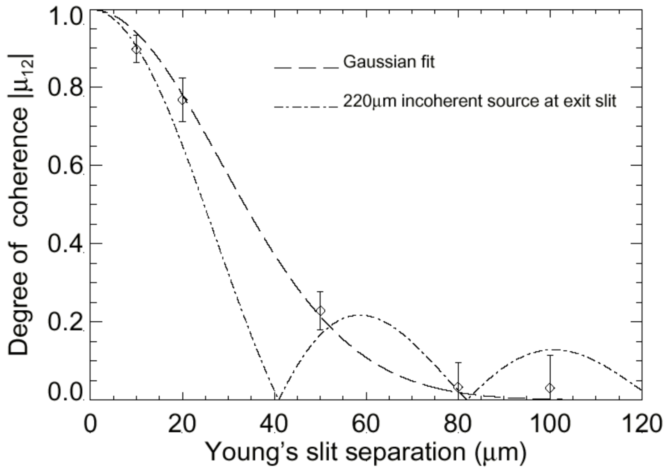
Young's slits (1.6 µm Au, 3 µm wide, 10 µm apart).



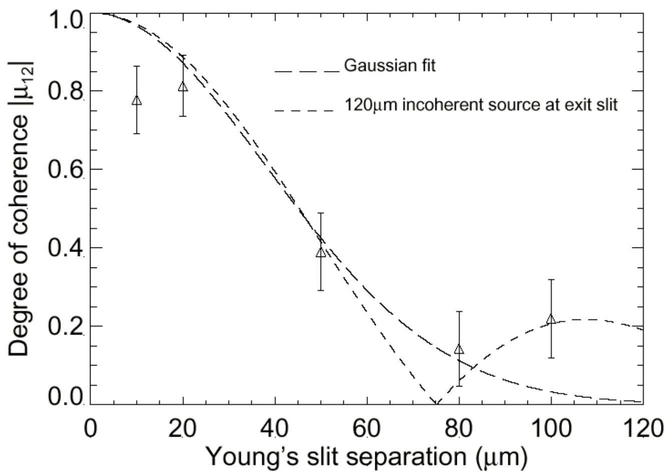
Young's fringes with 50 µm slit separation.

Coherence function at 2-ID-B

(a) Measured horizontal degree of coherence $|\mu_{12}|$ at 1.1 keV with monochromator entrance slit at 50 μm and exit slit at ~200 μm. Exit slit is 8.0 m from Young's slits. $|\mu_{12}|$ is dominated by beamline optics.



(b) Measured $|\mu_{12}|$ with exit slit at ~100 μm. $|\mu_{12}|$ is dominated by exit slit, producing sinc-like profile.



D. Paterson, et al., *Opt. Commun.* 195, 79 (2001)

Future plans in Sector 2

- Expand biological, environmental, and materials science applications of x-ray microscopy

- Increase performance of soft and hard x-ray microscopes
  - Push resolution to 30 nm and sensitivity to 1 ppb
  - Push strain and texture analysis to 1 µm scale
  - Improve user turnaround and ease of use

- Increase performance of fast x-ray tomography system
  - Push throughput to >1 3D data set/min
  - Push 3D resolution to 100 nm
  - Deliver turnkey operation

- Develop optics and microscopes for future x-ray sources

We are *2D*-brilliance driven

Coherent flux

Photons per time, spatial mode, and spectral bandwidth

$$F_c = B\,\lambda^2/4$$

Increasing access to undulators in Sector 2

X-ray microscopes

Diffraction-limited optics can only use a few coherent modes

>100 coherent modes in horizontal, <10 coherent modes in vertical direction

Hard x-ray beamline (2-ID-D/E) requires white-beam aperturing in horizontal

Soft x-ray beamline (2-ID-B) apertures pink beam after mirror deflection

Maximum utilization of and access to undulator beams

Rapid, easy ID branchline switching

Independent soft and hard x-ray undulators

Split ID beams with pick-off mirror

Also split ID sources?

Technical challenges to ID beam-splitting

Pick-off mirror (M2B)

Horizontally deflecting, horizontally focusing

Max power ~ 600 W, power density ~ 0.3 W/mm^2

Require <3 μrad figure error control

High power load near downstream edge

Edge quality must not adversely affect both soft, hard x-ray beams

Steering mirrror (M3B)

Horizontally deflecting, vertically focusing

Max power ~ 20 W, power density ~ 0.3 W/mm^2

Require <3 μrad figure error control

Must be thin (~1 cm) in order to pass hard x-ray beam to 2-ID-D/E

Current source limitations

- X-ray microscopes require < 1 µrad beam stability over days
  (few-µrad stability currently, except after refill during top-up,
  when itmay be as bad as 30 µrad in H or V planes)

- Most applications are brilliance-hungry, need 10^2 - 10^3 more!
  (but, can make 10-100x better use of B with better detectors)

- Large dead-time, dyn. range only ~ 10^6 with current fill pattern

- Undulator access is precious!

Optimum solutions

- ID improvements offer greatest brilliance gain!
- Reduced horizontal emittance is next-best solution to more B; want more symmetrical source (don't reduce vert. emittance)
- Many-bunch mode offers 10-50x more dynamic range in fast counting experiments (scanning transmission x-ray microscopy)
- Need full-time XBPM feedback to achieve needed stability
- Higher current buys us *very little.*